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Badische Anilin and Soda Fabrik AG. *DT-2149822-Q.
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1,4-OR 1,5-DICHLOROHYDROCARBONS - CONTINUOUS PREPN USING SIMPLE APPARATUS...

NEW

Continuous prepn. of 1,4- or 1,5-dichlorinated hydrocarbons by reaction of HCl with a liq. mixt. of 1,4- or 1,5-diols and/or corresponding cyclic ethers derived from opt. unsatd. hydrocarbons opt. with a catalyst. The vapour of the boiling reaction mixt. is fractionated such that a liq. mixt. of the dichloro cpd. and H_2O is obtd. on one hand and a gaseous mixt. of HCl, cyclic ether and opt. remaining dichloro cpd. on the other; after condensing, the gaseous mixt. is led back to the reactor mixt.

USE

Esp. for the prepn. of 1,4-dichlorobutane and 1,5-dichloropentane, this both cases the liq. mixt. of dichloro cpd. and H_2O is obtd. at a condensation temp. of 100-150°C at normal press.

ADVANTAGE

Only simple appts. required and normal or slightly increased press.

DETAILS

Apparatus consists of a boiling vessel fitted with a 2-part fractionating column. Between the upper and lower parts of the column is a take-off point for the liq. mixt. of dichloro cpd. and H_2O. The vapour mixt. of HCl and cyclic ether is taken off at the top of the column.

The process is esp. suited to cpds. with short chain alkyl grps. It is advantageous to use as catalyst hydrochlorides of tert. amines or quat. ammonium chlorides. The reactants are used in stoichiometric ratio or with an excess of HCl up to 50%. The reaction temp. is usually between 60-180°C, pref. 110-160°C and press. e.g. 0.5-3 atmos.

EXAMPLE

Into a mixt. of 15.8 kg. tributylamine hydrochloride, 3.6 kg. 1,4-butanediol and 0.4kg. H_2O warmed per hr. at 130°C, 1.3 kg 1,4-butanediol, 0.350 kg H_2O and 1.2 kg. HCl. The resulting vapour was passed through a 2-part distn. column, the lower part having 15 theoretical plates and the upper part 30. Between the 2 parts a magnetic condensate divider was provided for a reflux and a side-stream in ratio 1:1. The temp. of the liq. at take-off point was 100-105°C. Vapour temp. at head of column was 65-70°C. The vapours were condensed, 3-5 kg per hr. taken off and the rest recycled to the reaction flask. The condensate taken off at the side of the column was sepd. into a dichlorobutane layer and an aq. HCl layer, the latter being partially led back to the reactor.

Yield of 1,4-dichlorobutane - 93%.

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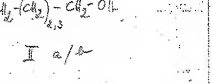

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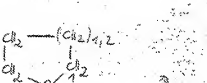

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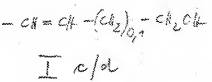

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